

Exhibit B

EFFECT OF DIFFERENT ALPHA-CELLULOSE LEVELS OF BIRCH PULP ON POLYNOSIC PROPERTIES

B. HACKMAN, R. SOILA and E. AVELA

Oy Kaukas Ab, Kaukas, Finland

This study was undertaken with a view to comparing birch Polynosic sulphite pulps of the same viscosity level, but with different alpha-cellulose contents, and to find out if it is possible to make a suitable dissolving pulp for Polynosic fibres using the acid magnesium bisulphite method instead of the calcium-base method.

Five pulps with cuprammonium viscosities of 44–48 cP were made in the laboratory, after which they were used in our viscose pilot plant for the preparation of viscose and for the spinning of Polynosic yarn. The physical characteristics of the fibres were tested with an Instron at the Finnish Pulp and Paper Research Institute. For the sake of comparison, viscoses were made under identical conditions from a commercial HWM-calcium sulphite

Table 1. Some characteristics of the pulps investigated in this study

| <i>Pulps</i> | <i>Mg-bisulphite (Lab.)</i> | | | | | <i>Ca-bisulphite (MII)</i> | <i>Prehydrolysed sulphate (MII)</i> |
|---------------------|---------------------------------|------|------|------|------|--------------------------------|---|
| Alpha-cellulose (%) | 90.1 | 91.1 | 92.6 | 93.2 | 94.6 | 93.4 | 98.0 |
| Beta-cellulose (%) | 4.3 | 3.6 | 3.9 | 3.6 | 2.4 | 1.8 | 2.1 |
| Gamma-cellulose (%) | 5.3 | 5.1 | 4.1 | 3.3 | 3.1 | 4.9 | 1.0 |
| Viscosity cuam (cP) | 45 | 48 | 45 | 47 | 45 | 44 | 23 |
| Acetone extract (%) | 0.16 | 0.14 | 0.23 | 0.13 | 0.25 | 0.23 | 0.09 |
| Penetration (%) | 6.1 | 9.0 | 4.4 | 4.2 | 3.7 | 4.0 | 1.5 |

birch pulp, and from a prehydrolysed hardwood sulphate pulp. The textile characteristics of the sulphate pulp mentioned have already been given in another paper (cf. page 289). In both investigations the spinning machine as well as the spinning conditions, were identical. The results obtained confirm, to a great extent, those presented in the above mentioned paper. *Table 1* shows some characteristics of the investigated *pulps*.

The alpha-cellulose contents of the magnesium bisulphite pulps ranged from 90.1 to 94.6 per cent, the alpha-cellulose content of the calcium base pulp was 93.4 per cent, and the prehydrolysed pulp had an alpha-cellulose

content of 96.0 per cent. The gamma-cellulose and pentosan figures decreased with increasing alpha-cellulose values, while in the beta-cellulose contents there are some irregularities. The beta- and gamma-cellulose contents of the calcium bisulphite pulp do not fit in the series. The low beta-cellulose content of this commercial calcium-base pulp is an advantage which can be seen from the physical properties of the fibres spun from it. The viscosities of the sulphite pulps were all within 44–48 cP.

Table 2. Viscose preparation conditions

| | | | | | |
|-------------|-----------------|---------|----------------------|------------------------------|-------------|
| Steeping: | NaOH | 19% | Ripening: | Time | 21 h |
| | Time | 60 min | | Temperature | 12–20°C |
| | Temperature | 19°C | | | |
| Shredding: | Time | 30 min | Filtrations: | 1. Linen + linters + linen | |
| | Temperature | 19°C | | 2. Linen + linters + flannel | |
| | | | | + linen | |
| Ageing: | Time | 3–4 h | | 3. Linen + linters + nylon | |
| | Temperature | 28°C | Viscose composition: | Cellulose | 5.0% |
| Xanthation: | CS ₂ | 55% | | NaOH | 3.5% |
| | Time | 2.5 h | | Modifiers | 0.15% |
| | Temperature | 26°C | Viscose properties: | Viscosity | 200–400 sec |
| Dissolving: | Time | 5 h | | Gamma number | 62 |
| | Temperature | 10–12°C | | | |

The steeping was carried out on samples of 650 g pulp with hemifree sodium hydroxide of 19 per cent strength (Table 2). The ageing time for all sulphite pulps was 3–4 h, the sulphate pulp needed no ageing. The rotating speed of the dissolver was 1000 rev./min. All viscose preparation stages were temperature controlled. The ripening of the viscose took place under vacuum overnight and the spinning was started the following morning. Small amounts of modifiers improved the spinnability of the viscoses.

Figure 1 shows a continuous type of spinning machine which has been converted for Polynosic spinning. It allows a stretching of yarn up to 200 per cent. In the place of the original stretching bath a new trough was constructed in which both the spinning and stretching were carried out.

Figure 2 shows the details of the stretching arrangements. The filaments coming from the spinneret advance between an eight stepped wheel and eight independent rotating guide rollers. When leaving the largest wheel the yarn has been extended three times. The distance of stretching is 320 cm. The spinning conditions used are given in Table 3.

After regeneration with sulphuric acid the yarn was washed with water on four process reels, treated with a rayon finishing agent and dried. No bleach was used.

The results of these spinning experiments are given in Table 4. The results are divided into two groups, one for the spinnings, with viscose viscosities of 200–250 sec ball fall and another for viscosities between 300–360 sec ball fall. One of the magnesium-base pulps is represented in both viscosity groups.

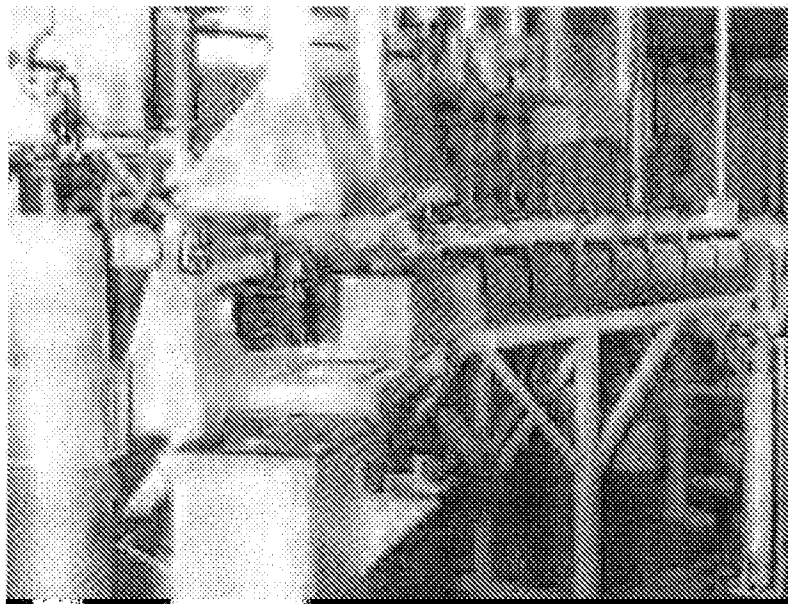


Figure 1. Polynosic spinning machine



Figure 2. Stretching arrangements used in making Polynosic rayon

Table 3. Spinning conditions used

| | |
|--------------------------------|---|
| Spinning bath composition: | $\begin{cases} \text{H}_2\text{SO}_4 & 16.0 \text{ g/l} \\ \text{Na}_2\text{SO}_4 & 10.0 \text{ g/l} \\ 2\text{Zn SO}_4 & 0.2 \text{ g/l} \\ \text{Additive} & 1.0 \text{ g/l} \end{cases}$ |
| Spinning bath temperature: | 25°C |
| Stretching: | 200% in the spinning bath |
| Regenerating bath composition: | H_2SO_4 22.0 g/l |
| Regenerating bath temperature: | 70°C |
| Relaxation: | 1% |
| Spinning speed: | 25 m/min |
| Total denier: | 790 |
| Spinnerets: | 720/0.05 |

The left part of Table 4 shows a correlation between the wet strength, wet/dry tenacity ratio and alkali solubility on one hand and the alpha-cellulose content on the other hand. The alpha-cellulose content of the pulp also seems to influence the loop tenacity to some extent. The good tenacity figures of the calcium bisulphite pulp might depend on its low beta-cellulose content (1.8 per cent). On the right is the high viscosity part of the table. Some of the textile characteristics are on different levels from those on the left (the low viscosity part of the table). In the 300–360 viscosity group, the conditioned tenacities are lower but the figures of the tenacity ratio and the loop tenacity are higher than in the 200–250 viscosity group. In the high

Table 4. Properties of fibres

| Pulp | Viscose viscosity: 200–250 | | | | | Viscose viscosity: 300–360 | | |
|--|----------------------------|------|---------------|--------------------------|------|----------------------------|------|------|
| | Mg bisulphite | | Ca bisulphite | Phosphoric acid sulphate | | Mg bisulphite | | |
| Alpha-cellulose content (%) | 90.1 | 92.6 | 92.6 | 92.4 | 98.0 | 90.1 | 91.1 | 92.2 |
| DP | 604 | 600 | 615 | 614 | 614 | 660 | 665 | 660 |
| Conditioned tenacity (g/den.) | 5.16 | 5.65 | 6.34 | 6.56 | 6.42 | 4.81 | 4.79 | 5.14 |
| Wet tenacity (g/den.) | 3.93 | 4.16 | 4.39 | 4.52 | 4.61 | 4.18 | 4.00 | 4.25 |
| Ratio T_w/T_d (%) | 76 | 74 | 69 | 69 | 85 | 86 | 84 | 84 |
| Conditioned elongation (%) | 6.9 | 7.8 | 8.8 | 7.3 | 7.8 | 7.3 | 6.9 | 6.6 |
| Wet elongation (%) | 8.4 | 9.2 | 9.7 | 8.6 | 9.8 | 8.7 | 8.4 | 8.8 |
| Loop tenacity (g/den.) | 0.61 | 0.57 | 0.66 | 0.76 | 0.80 | 0.83 | 0.91 | 0.72 |
| Wet modulus at 5% strain (g/den.) | 44.9 | 42.8 | 47.4 | 46.6 | 46.6 | 42.8 | 46.3 | 47.1 |
| Alkali solubility in 5% NaOH (%) | 2.8 | 2.5 | 2.2 | 2.1 | 2.1 | 2.6 | 3.6 | 2.6 |
| Wet tenacity after treatment with 5% NaOH (g/den.) | 3.33 | 3.61 | 3.87 | 3.82 | 4.14 | 3.19 | 3.38 | 3.73 |

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viscosity part of the table, the wet modulus follows well the increase of the alpha-cellulose content of the pulp. The viscose preparation conditions seem to have a strong influence on the loop tenacity values. It is obvious that the high level of the loop tenacity is connected to the high viscosities or DP-values.

In Figure 3, some of the values of the lower viscosity part of Table 4 are plotted against the alpha-cellulose content.

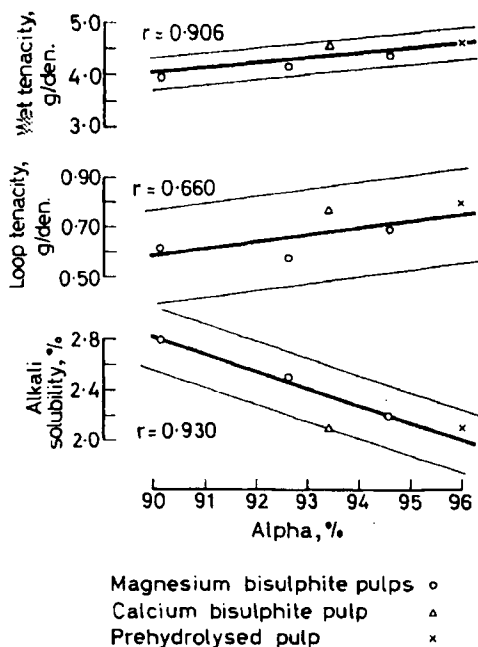


Figure 3. Wet tenacity, loop tenacity and alkali solubility of Polynosic fibres plotted against the alpha contents of the pulps [O, Magnesium bisulphite pulps; Δ, calcium bisulphite pulp; x, prehydrolysed pulp]

The inclination of the line drawn through the points of the wet tenacities shows that when the alpha-cellulose content of the pulp increases by 1 per cent, the wet tenacity increases by 0.1 g/den., which is identical to the result mentioned in the other paper (cf. page 289). The loop tenacity has a poor correlation to the alpha-cellulose content but, contrary to this, the decreasing alkali solubility of the fibre follows well the increase of the alpha-cellulose level of the pulp.

The five points in each diagram have been treated statistically and in Figure 3 we see the regression lines ± 2 times the standard deviation of the single values about the regression line. In each case r is the regression coefficient.

At the conclusion of these experiments it can be stated that it is possible to produce fibres, with Polynosic properties, from acid magnesium bisulphite

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pulps as well as from other bisulphite pulps or from prehydrolysed pulps. In addition to this it was noticed that the viscose viscosity and the degree of polymerization have a great influence on the results, especially in the manner that higher viscose viscosity gives a higher wet tenacity to conditioned tenacity ratio and a higher loop tenacity.